Dimethyl methylphosphonate-based nonflammable electrolyte and high safety lithium-ion batteries

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Abstract

Dimethyl methylphosphonate (DMMP) was used as a cosolvent to reformulate the nonflammable electrolyte of 1 M $LiPF_6$/EC + DEC + DMMP (1:1:2 wt.) in order to improve the safety characteristics of lithium-ion batteries. The flammability, cell performance, low-temperature performance and thermal stability of the DMMP-based electrolyte were compared with the electrolyte of 1 M $LiPF_6$/EC + DEC (1:1 wt.). The nonflammable electrolyte exhibits good oxidation stability at the $LiCoO_2$ cathode and poor reduction stability at the mesocarbon microbead (MCMB) and surface-modified graphite (SMG) anodes. The addition of vinyl ethylene carbonate (VEC) to the DMMP-based electrolyte provided a significant improvement in the reduction stability at the carbonaceous electrodes. Furthermore, it was found that the addition of DMMP resulted in optimized low-temperature performance and varied thermal stability of the electrolytes. All of the results indicated the novel DMMP-based electrolyte is a promising nonflammable electrolyte to resolve the safety concerns of lithium-ion batteries.
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1. Introduction

Lithium-ion batteries are now widely used as energy storage devices for portable electronic devices such as laptop computers, cellular phones and digital cameras due to their high-energy density. Large-sized lithium-ion batteries and battery packs are also very attractive for electric vehicles (EV) and hybrid electric vehicle (HEV) applications. However, safety concerns have become the most important issue for the development of lithium-ion batteries, especially for the high-energy battery packs, because under abusive conditions, lithium-ion batteries may undergo thermal runaway that generates a sharp rise in temperature and then results in serious hazards of fire and explosion [1–3].

Much effort has been devoted to improve the safety characteristics of lithium-ion batteries. Most of the separator membranes such as the polyolefin products have been found to exhibit shut-down performance, i.e. the polymer chains can swell with the temperature rise so that the pores shrink and shut down the cell by turning a porous ionically conductive polymer membrane into a non-porous insulating layer between the electrodes [4,5]. Some redox shuttle and electrochemical polymerisable additives are found to be effective in preventing overcharge [6,7]. In addition, the use of gel polymer electrolytes is also expected to achieve safety improvement [8]. Nevertheless, the heat generation under the abuse conditions cannot be prevented if using the approaches above to solve the safety problem. As far, there seems to be no efficient measure to totally inhibit the thermal runaway. So the flammable carbonate solvent in the state-of-the-art nonaqueous electrolytes might be ignited inevitably, even though those measures above are made to improve the safety characteristics of lithium-ion batteries. In recent years, many researches have been focused on the development of fire retardant (FR) additives to lower the flammability of the nonaqueous electrolytes [9–16].

The reformulation of the electrolytes by adding the FRs into the common electrolytes can result in nonflammability or at least retard flammability of the whole electrolyte systems, which

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seems to be a more efficient solution on the thermal safety of the lithium-ion batteries. In most cases, the amount of FRs in the electrolytes is kept at a minimum, usually no more than 20% either by weight or by volume, for any compromise in the cell performance [11–14]. However, the so-called nonflammable electrolytes as well as the flame-retarded electrolytes, with the limited amount of FRs, are still capable to be ignited under some special conditions, for example at the atmosphere with high concentration of oxygen gas, or being caught fire continually. As a straightforward solution to this issue, the nonflammable electrolyte containing flame retardant additives/cosolvents with a higher concentration, for example no less than 50% by weight, can easily exhibit better safety characteristics not only for itself, but also can suppress the flammability of other materials contacted. Wang et al. reported high concentration (exceeding 70%) trimethyl phosphate (TMP)-based nonflammable electrolytes [17]. They concentrated the effort on improving the compatibility of the TMP-based electrolytes with graphitic anode materials, but little was mentioned in detail about the safety characteristics.

In our recent work [18], we reported an efficient flame retardant additive for the electrolyte—dimethyl methylphosphonate (DMMP) with the advantages of moderate viscosity (1.75 mPa s), high dielectric constant (22.3), low melting point ($-50\,°C$) and high boiling point ($180\,°C$). Taking these properties combined with its high flame retardant efficiency into account, we prepared a totally nonflammable electrolyte for the batteries, with the high concentration of DMMP (50 wt.%), in order to explore the high safety electrolyte and lithium-ion batteries. In this paper, we explored the feasibility of DMMP-based electrolyte as the high safety lithium-ion battery electrolyte. Besides the thermal safety characteristics, the compatibility of the novel electrolyte with electrodes, including the $LiCoO_2$ electrode and carbonaceous anodes, and the electrochemical performance at low temperatures were also investigated and discussed.

2. Experimental

As-purchased DMMP (Qingdao Lianmei Chemical Co. Ltd.) was purified with a distillation step under vacuum and dried before use over molecular sieves (4A). Carbonate solvents (Guotai Huarong New Chemical Materials Co.) and $LiPF_6$ (Tianjin Jinniu Power Sources Material Co.) were used as received. Vinyl ethylene carbonate (VEC) (Ferro, battery-grade) was used to improve the solid electrolyte interface layer between the electrolyte and the anode. The electrolytes were prepared under a highly pure argon atmosphere in an argon-filled glove box (MBrun Labmaster 130). All solvent ratios indicated in this paper were in weight ratios. A positive electrode consisting of $LiCoO_2$ (84 wt.%), acetylene black (8 wt.%) and poly(vinylidene fluoride) (PVDF) (8 wt.%) and a negative electrode consisting of carbonaceous materials (92 wt.%) and PVDF (8 wt.%) were made on aluminum and copper foils, respectively. The mass loading of both the two electrodes was about 7.5 mg cm^{-2}. Two kinds of carbonaceous materials, MCMB (mesocarbon microbeads) graphite and a surface-modified graphite (SMG), were chosen in this study. The SMG powder

was prepared by a thermal vapor decomposition treatment [19]. It can also be prepared by a pyrolytic polymer encapsulation [20] or a hydrothermal treatment [21]. CR2032 coin cells were assembled in the glove box and then cycled on a multi-channel battery cycler (Neware BTS2300, Shenzhen). In the $LiCoO_2/C$ full-cells, the mass ratio of $LiCoO_2$ and carbonaceous materials was controlled at about 1:1, so that the cell capacity was determined by the positive electrode.

Ionic conductivities of the electrolyte solutions were measured over a wide temperature range of -40 to $55\,°C$ using a Model DDS-307A conductometer (Shanghai Precision & Scientific Instrument Co. Ltd., China). Each measurement of conductivity at a given temperature was carried out after holding the electrolyte solution at the temperature for 30 min in order to reach full thermal equilibration. The temperature of the electrolyte solution and subsequent cell testing at low temperatures was controlled by a WD4005 low-temperature chamber (Shanghai Experimental Equipment Co. Ltd., China).

For $LiCoO_2/C$ full lithium-ion cells and $LiCoO_2/Li$ positive half-cells, the cycling tests were performed at a current of 0.2 mA cm^{-2} in the constant current–constant voltage (CC–CV) charge mode and constant current (CC) discharge mode. The constant voltage (CV) regime ended until the current was less than 0.03 mA cm^{-2}. The C/Li negative half-cells were cycled between 3.0 and 0.01 V at a current density of 0.20 mA cm^{-2}. The batteries used for low temperature tests were first charged at room temperature at the same CC–CV charging protocol, and then held at the controlled low temperature for 30 min before discharging at a current of 0.1 mA cm^{-2}.

The electrochemical stability of the electrolytes on the negative electrodes was measured by cyclic voltammetry (CV) between 0 and 3.0 V at a scan rate of 0.2 mV s^{-1}. The ac impedance was measured with the frequency range and voltage amplitude set as 10 kHz–0.01 Hz and 5 mV, respectively. Both measurements were carried out on these cells with a CHI 604A Electrochemical Workstation.

We measured the self-extinguishing time (SET) and limited oxygen index (LOI) to evaluate the flammability of the electrolytes. The detailed procedures were described elsewhere [10,18]. The thermal stability of different electrolytes was evaluated by using a Calvet-type calorimeter (Setaram C80). About 0.25 g electrolyte was placed in a high-pressure stainless steel vessel with dry argon atmosphere. The measurement was performed in the temperature range from ambient temperature to $300\,°C$ at a $0.2\,°C$ min^{-1} heating rate.

3. Results and discussion

3.1. Flammability and thermal stability

In our previous study [18], we used limited oxygen index (LOI) as well as the self-extinguish time (SET) to evaluate the flammability of the electrolytes. Here nonflammability of the novel electrolyte of 1 M $LiPF_6$/EC + DEC + DMMP (1:1:2) was also evaluated by these two parameters, compared with the baseline electrolyte of 1 M $LiPF_6$/EC + DEC (1:1), as shown in Fig. 1. Obviously, the baseline electrolyte is very flammable with SET

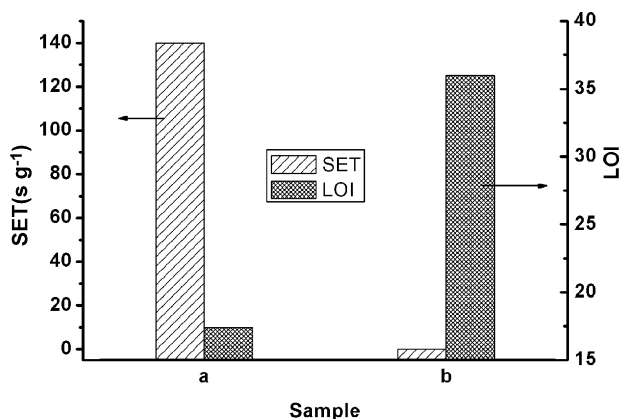Fig. 1. The results of flammability test of SET and LOI for 1 M LiPF$_6$/EC + DEC(1:1) (a) and 1 M LiPF$_6$/EC + DEC + DMMP(1:1:2) (b).

Table 1
Comparison of heat generation in some temperature sections of three kinds of electrolytes (a: 1 M LiPF$_6$/EC + DEC(1:1), b: 1 M LiPF$_6$/EC + DEC + DMMP(1:1:2), c: 1 M LiPF$_6$/EC + DEC + DMMP(1:1:2) + 5 wt.% VEC)

	Heat (J g^{-1})		
	70–140 °C	140–220 °C	220–300 °C
a	32.8	363.2	467
b	32.8	173.1	925
c	29.7	125.9	998

of 140 s g^{-1} and LOI of 17. Once DMMP is used as the cosolvent in the electrolyte, the SET greatly decreases to 0 s g^{-1}, that is, the DMMP-based electrolyte cannot be ignited in the testing conditions. The nonflammability has also been validated by the LOI test more obviously since the DMMP-base electrolyte has a high LOI of 36, which is seldom reached even for the flame-retarded plastic materials. Therefore, using high concentration DMMP as cosolvent for the electrolyte in the lithium-ion batteries, we can easily formulate a totally nonflammable electrolyte. Above all, it can be believed that people will seldom find fire and explosion accidents concerning lithium-ion batteries with this kind of nonflammable electrolyte.

Fig. 2 shows C80 data for three kinds of electrolytes, including the baseline electrolyte, the DMMP-based electrolyte containing VEC and not, respectively. From the results given by C80, the heat generation calculated in some temperature sections are shown in Table 1. It can be seen that the DMMP-containing electrolytes show higher onset temperature of the major exothermal reaction around 200 °C, especially when VEC is added. As the introduction of DMMP, the contents of carbonates are decreased substantially so that the exothermic reactions associated with EC and DEC are suppressed in a lower level, with less
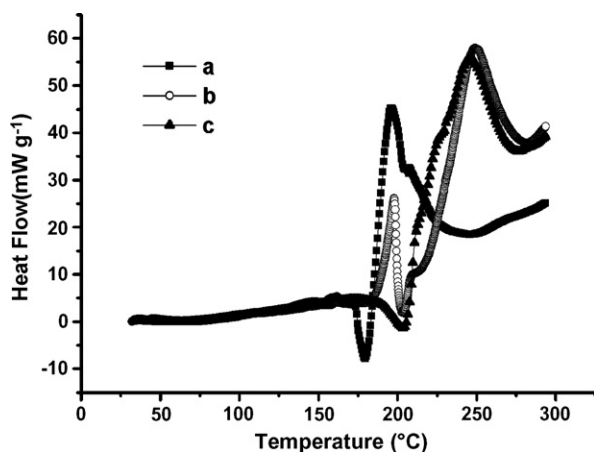
heat released before 220 °C. Nevertheless, between the range of 220 and 300 °C, the heat generation for the electrolytes containing DMMP is much more than that for the baseline electrolyte. The most possible reason is that DMMP could decompose at the high temperature (>220 °C) and further produce some particles which may catalyze the reactions associated with the carbonates. Although the results of these thermal investigations are inadequate to evaluate the thermal stability of electrode/DMMP-based electrolyte systems, they still show that the addition of DMMP in the electrolytes largely has suppressed the heat-flow in the temperature range from room temperature to 220 °C, which is helpful to achieve better thermal stability of a lithium-ion cell to some degree.

3.2. Ionic conductivity

Ionic conductivity of the nonflammable electrolyte was compared with the baseline electrolyte in the temperature range of −40 to 55 °C. Fig. 3 shows the dependence of ionic conductivity on temperature for the DMMP-based and baseline electrolytes. It can be seen obviously that both conductivity of the two kinds of electrolytes monotonically decreases with the temperature declining, and the conductivity of DMMP-based electrolyte is higher than the baseline electrolyte at the same temperature, especially at relatively low temperature (≤−20 °C). It is worth mentioning that in the baseline electrolyte a solid phase was observed to precipitate from the solution below −25 °C; at that
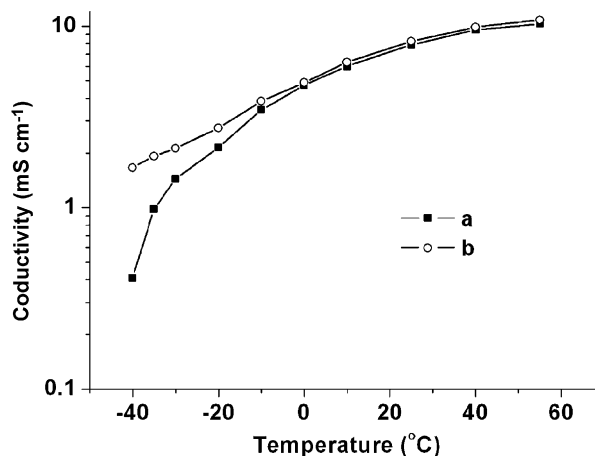

Fig. 2. C80 heat-flow profiles of three kinds of electrolytes (a: 1 M LiPF$_6$/EC + DEC(1:1), b: 1 M LiPF$_6$/EC + DEC + DMMP(1:1:2), c: 1 M LiPF$_6$/EC + DEC + DMMP(1:1:2) + 5 wt.% VEC).

Fig. 3. The dependence of ion conductivity on temperature for the baseline electrolyte (a: 1 M LiPF$_6$/EC + DEC(1:1)) and DMMP-based electrolyte (b: 1 M LiPF$_6$/EC + DEC + DMMP(1:1:2)).

moment, the conductivity measured only reflects on the ion transport in the liquid phase. However, for the nonflammable electrolyte, until −40 °C only a little amount of suspended particles can be found. We speculate that the higher ionic conductivity of the nonflammable electrolyte maybe originate from the low viscosity and high dielectric constant of DMMP. Since it is known that a high dielectric constant favors the dissociation of ion pairs from the dissolved salt and that low viscosity accelerates ionic movement, the simultaneous effect of both factors as the consequence of DMMP presence will lead to the conductivity rise of the nonflammable electrolyte, which is certainly a desired property for their performance in cells.

When the temperature falls to −25 °C, the conductivity of the baseline electrolyte system begins to drop sharply, and close to zero at −40 °C due to the freezing of the electrolyte, whereas nonflammable electrolyte system still exhibits considerable conductivity even at −40 °C. As we all know, EC in both electrolytes has a relatively high freezing point of 37 °C, which drops sharply when mixed with the other solvent [22]. Furthermore, the dissolution of $LiPF_6$ can further bring down the phase transition temperatures of the mixed solvent solutions. Thus, it is easy to understand that the nonflammable electrolyte with lower concentration of high freezing component EC and higher concentration of low freezing component DMMP exhibits a lower freezing temperature, which results in a better low-temperature performance. From the consideration of ion conductivity, it can be concluded that the introduction of a great deal of DMMP should be favorable, especially at relatively low temperatures.

3.3. Electrochemical performance in the LiCoO₂/Li half-cells

Fig. 4 shows the electrochemical performance of $LiCoO_2$/Li half-cells with the nonflammable electrolyte compared with the baseline electrolyte at the room temperature. It can be seen that there is no significant difference between the two kinds of electrolytes for the first charge–discharge cycle voltage profiles (Fig. 4a). Therefore, the DMMP-based electrolyte should be stable on the $LiCoO_2$ positive electrode, and the achievement of similar specific capacity is consistent with the ionic conductivity between these electrolytes. However, the cycling performance of the $LiCoO_2$/Li cells with the nonflammable electrolyte seems better than the baseline electrolyte (Fig. 4b). We believe that DMMP may be beneficial for the surface chemistry on the interface between the cathode and the electrolyte, just like our previous work using trimethyl phosphite as an additive [23].

The low-temperature performance of lithium-ion cells is mainly limited by the electrolyte. Fig. 5 shows the typical temperature dependence of the voltage profiles of two $LiCoO_2$/Li cells using the nonflammable electrolyte and the baseline electrolyte. As the ambient temperature decreases, it is observed that the total energy output decreases. The capacity utilization accessed at −20 °C and above is nearly constant for all the cells with both of the electrolytes tested, although the conductivities change markedly with the temperature (Fig. 3). It seems that the precipitation of either lithium salt or the high-melting solvent component (EC) occurs between −20 and −30 °C for
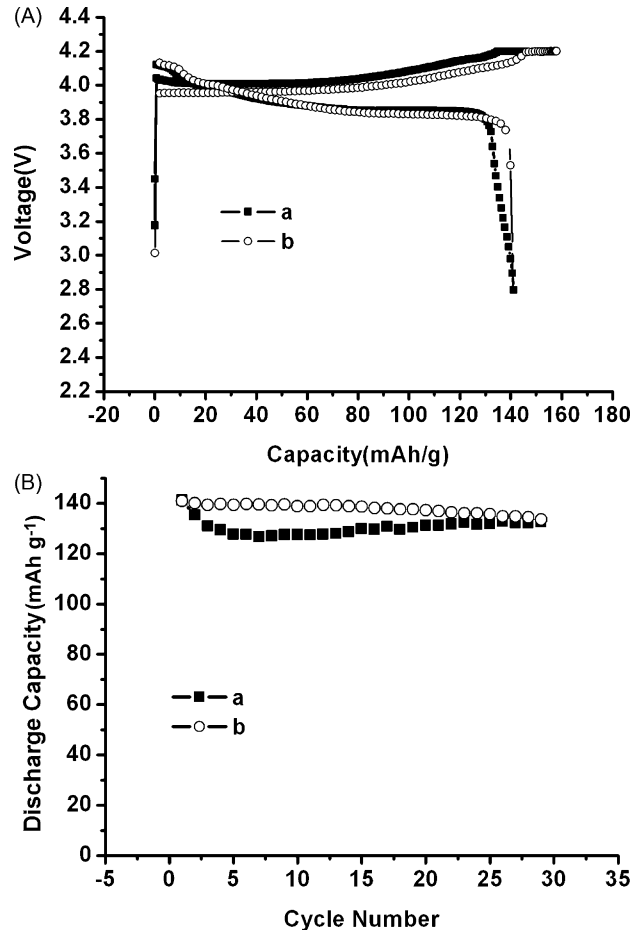

Fig. 4. The first charge–discharge voltage profiles (A) and cycling performance (B) of $LiCoO_2$/Li half-cells with the baseline electrolyte (a: 1 M $LiPF_6$/EC + DEC(1:1)) and DMMP-based electrolyte (b: 1 M $LiPF_6$/EC + DEC + DMMP(1:1:2)).

the baseline electrolyte, so that the discharge capacity sharply decreases and only reached about 12 mAh g⁻¹ at −35 °C. As for the DMMP-based electrolyte, no precipitation can be found and the overall discharge capacity is 90 mAh g⁻¹ at −35 °C for the
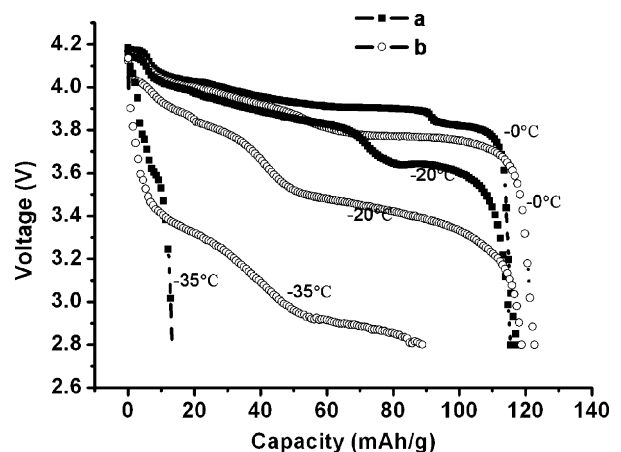

Fig. 5. Discharge capacities of $LiCoO_2$/Li half-cells at the different temperatures with 1 M LiPF6/EC + DEC(1:1) (a) and 1 M LiPF6/EC + DEC + DMMP(1:1:2) (b).

LiCoO$_2$/Li half-cell, much more than that of the baseline electrolyte. The depression in cell voltage at low temperatures has been aggravated by DMMP as cosolvent, perhaps because of the violently increased resistance in electrolyte/electrode interfaces besides in bulk electrolyte. Therefore, a fortunate fact is that the nonflammable electrolyte containing 50 wt.% DMMP not only possesses the character of nonflammability, but also achieves the all-right low-temperature performance.

3.4. Electrochemical stability on the anode materials

Our previous study has disclosed that DMMP may lead to the exfoliation of an untreated graphite negative electrode at a low potential versus Li/Li$^+$, and a surface-modified graphite (SMG) is acceptably compatible with the electrolytes containing low concentration of DMMP (≤20%) [18]. Here, we probe the compatibility of two different carbonaceous anodes – MCMB and SMG – with the electrolyte containing relatively high concentration of DMMP (50 wt.%) in detail. Fig. 6 shows the results of the electrochemical performance of three kinds of electrolytes

on the MCMB and SMG electrodes, respectively. The approximate theoretical capacities of electrodes can be reckoned on the baseline electrolyte used. DMMP in the nonflammable electrolyte has similar stereo hindrances to propylene carbonate and would exert similar strains into the graphite structure and cause exfoliation. VEC is confirmed as an efficient film formation additive in forming a stable SEI on the graphite surface that protects against PC decomposition and graphite exfoliation [24]. With addition of 5 wt.% VEC to the DMMP-based electrolyte, reversible lithium intercalation/deintercalation occurs. Cyclic voltammetry of the three electrolytes on the carbonaceous anodes, as shown in Fig. 7, indicates that the reduction of DMMP occurs at ∼1.2 V, and is eliminated after VEC reduces at ∼1.5 V. There seems no obvious difference with the different electrodes for the same electrolyte, except for the coulombic efficiency that is 85.8% for MCMB and 88.2% for SMG. Compared with the baseline electrolyte, VEC in 5 wt.% presence successfully eliminates the reduction of DMMP on the SMG electrode, but not entirely on the MCMB electrode. It must be mentioned that the addition of VEC distinctly results in the loss
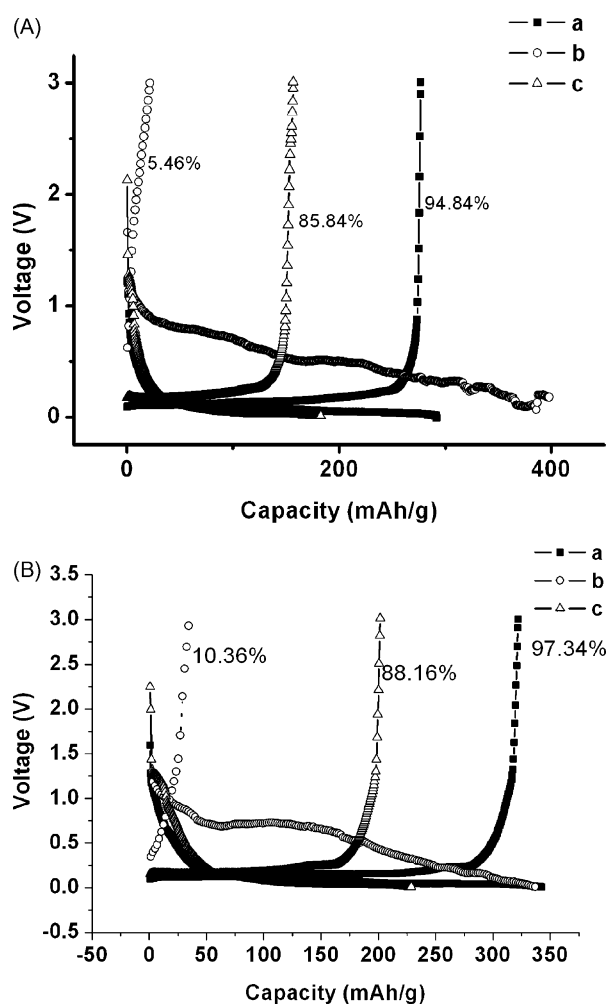

Fig. 6. The first charge–discharge voltage profiles of MCMB/Li (A) and SMG/Li (B) half-cells with three kinds of electrolytes (a: 1 M LiPF$_6$/EC + DEC(1:1), b: 1 M LiPF$_6$/EC + DEC + DMMP(1:1:2), c: 1 M LiPF$_6$/EC + DEC + DMMP (1:1:2) + 5 wt.% VEC).
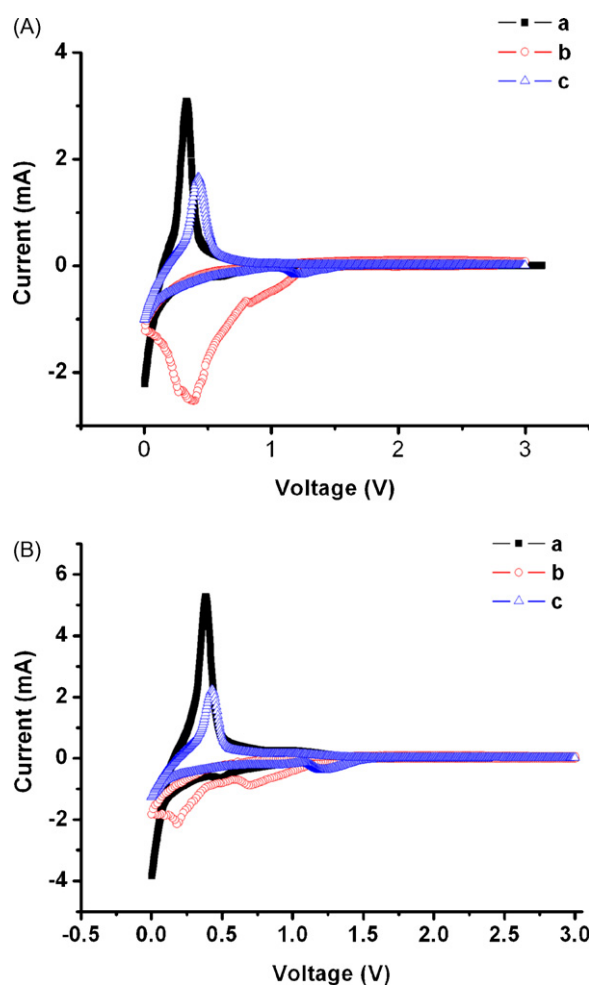

Fig. 7. Cyclic voltammetry of MCMB/Li (A) and SMG/Li (B) half-cells with three kinds of electrolytes (a: 1 M LiPF$_6$/EC + DEC(1:1), b: 1 M LiPF$_6$/EC + DEC + DMMP(1:1:2), c: 1 M LiPF$_6$/EC + DEC + DMMP(1:1:2) + 5 wt.% VEC).
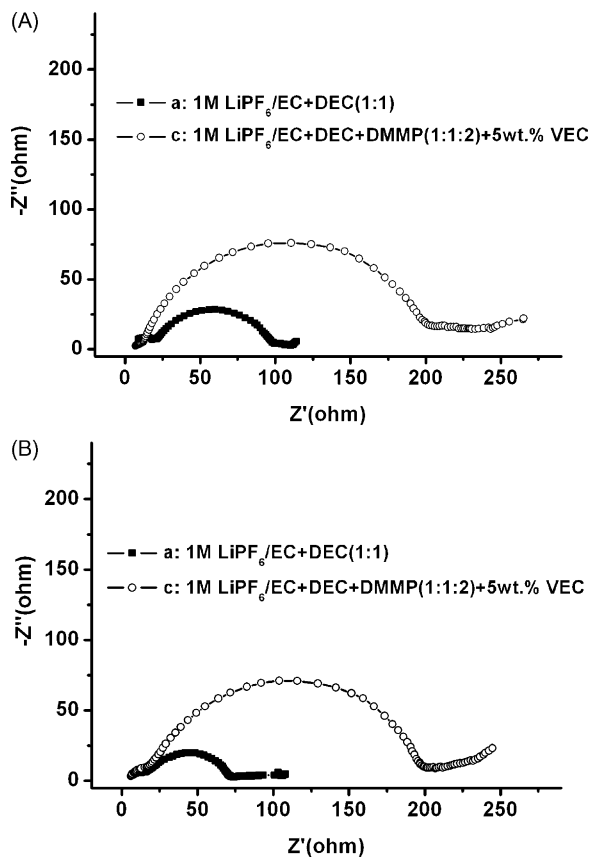
Fig. 8. ac impendence of MCMB/Li (A) and SMG/Li (B) half-cells with two kinds of electrolytes (a: 1 M LiPF$_6$/EC + DEC(1:1), c: 1 M LiPF$_6$/EC + DEC + DMMP(1:1:2) + 5 wt.% VEC).

of the specific capacity (Fig. 6), because of the substantial passivated layer with a high impedance, as shown in Fig. 8. This impedance rise should be contributed by the formation of passivated layers on the carbonaceous electrode as well as on the lithium foil electrode in these half-cells because VEC can be reduced at a low potential approaching that of metallic lithium. Considering the fact that the potential on the lithium side is always low, it is reasonable to believe that the passivated layer on the lithium electrode is likely thicker than on the carbonaceous electrode.

3.5. Performance in full-cells

To evaluate the practicability of the DMMP-based electrolyte, Fig. 9 shows the electrochemical performance of the LiCoO$_2$/MCMB and LiCoO$_2$/SMG full-cells with the nonflammable electrolyte containing 5 wt.% VEC. Discharge capacity is calculated based on the mass of LiCoO$_2$. It can be seen that the nonflammable electrolyte in the LiCoO$_2$/SMG full-cell leads to better cycle performance in the first 50 cycles with the capacity loss of 0.36% every cycle, compared with the 0.61% every cycle for the LiCoO$_2$/MCMB full-cell. The nonideal capacity loss in the LiCoO$_2$/MCMB full-cell may be caused because 5%VEC content of the electrolyte is not enough to eliminate the reductive decomposition of DMMP completely, which suggests simultaneously that SMG is the more suitable
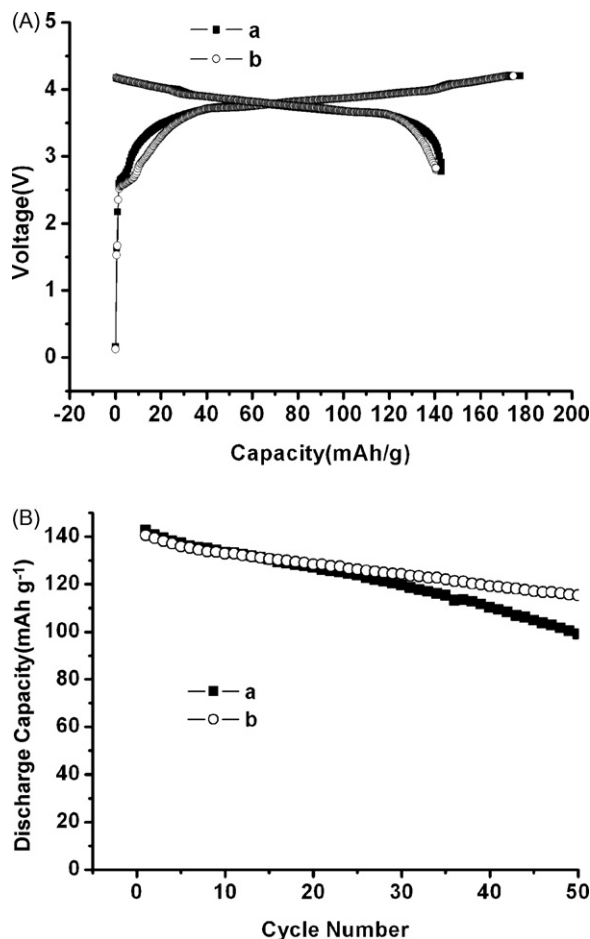

Fig. 9. The first charge–discharge voltage profiles (A) and cycling performance (B) of LiCoO$_2$/MCMB (a) and LiCoO$_2$/SMG (b) full-cells with the DMMP-based electrolyte containing 5 wt.% VEC (1 M LiPF$_6$/EC + DEC + DMMP(1:1:2) + 5 wt.% VEC).

anode material in the DMMP-based electrolyte. Here, the voltage is high enough to avoid the film-forming additive (VEC) being reduced to form the impact layer on the LiCoO$_2$ electrode, which is different from the lithium foil in the Li/C half-cells (Fig. 8). Therefore, the interfacial impedance increased by the introduction of VEC seems to have no distinct effect on the cycling of the full-cell. According to these results above, we believe that the DMMP-based electrolyte would be chosen in practice if the film formation additive were optimized further.

4. Conclusions

DMMP was used as a main component of the nonflammable electrolyte, which had the higher ion conductivity than the state-of-the-art electrolyte, especially at low temperature. The nonflammable DMMP-based electrolyte exhibited excellent stability with LiCoO$_2$ cathode but poor stability against carbonaceous anodes. Fortunately, VEC – the film formation additive – could efficiently improve the compatibility of the nonflammable electrolyte with MCMB and SMG electrodes, although capacity was reduced because of the increasing impedance of the interfaces between the electrolyte

and electrode. Through reviewing the LiCoO$_2$/MCMB and LiCoO$_2$/SMG full-cells, we found that the nonflammable electrolyte modified by VEC exhibited the acceptable capacity loss, especially for the latter. The introduction of DMMP also could improve the thermal stability of the electrolyte in the temperature range below 220 °C, and widen the temperature range of service for lithium-ion cells. In brief, the DMMP-based electrolyte exhibits significant nonflammability and provides us with a promising approach to the high safety lithium-ion batteries.

Acknowledgements

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